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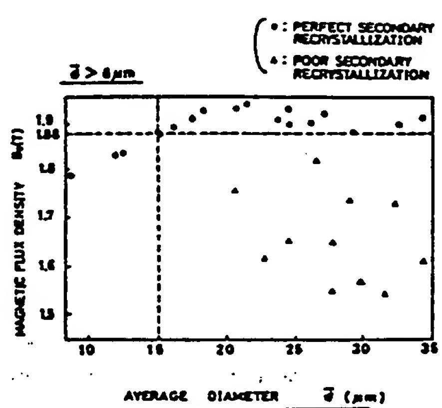
(54) **A method of manufacturing a grain-oriented electrical steel strip.**

(57) Provided is a method of manufacturing a grain-oriented electrical steel strip having excellent magnetic properties, that is, high magnetic flux density in particular, which is characterized in that the average diameter $\bar{d}$ of grain (primary recrystallization grain) of a material before final annealing is set to a value greater than 15 µm and the coefficient σ^* of deviation in grain diameter is set to a value less than 0.6.

According to the present invention, the aggregation to the {110}<001> orientation of a product can be made to be remarkably high so as to obtain a product having a high magnetic flux density B_8 which is equal to or greater than 1.88 tesla.

FIG. 1



EP 0 378 131 A2

A METHOD OF MANUFACTURING A GRAIN-ORIENTED ELECTRICAL STEEL STRIP

Field of the Invention:

The present invention relates to a method of manufacturing a grain-oriented electrical steel strip adapted to be used as an iron core in a power supply transformer and a large size rotary machine. In more detail, the present invention relates to a method of manufacturing a grain-oriented electrical steel strip having an extremely high magnetic flux density by controlling the average value of grain sizes and the deviation of primary recrystallization grain to desired values in a grain-oriented electrical steel strip manufacturing process.

Background of the Invention:

A grain-oriented electrical steel strip is used as an iron core in a power supply transformer or a large size rotary machine, and is required to have excellent magnetic properties such as excitation property, a core loss property and the like. The magnetic property is generally exhibited by a magnetic flux density (B_8 value) at a magnetic field intensity of 800 A/m. Meanwhile, the core loss property is exhibited in general by a power loss $W_{17/50}$ (W/kg) per Kg of an iron core which is magnetized up to 1.7 tesla at a frequency of 50 Hz. The magnetic flux density of the grain-oriented electrical steel strip is a very important property, that is, the higher the magnetic flux density the more satisfactory the core loss property becomes (the core loss value is decreased).

On the contrary, a grain-oriented electrical steel strip having a high magnetic flux density has in general a large grain size of secondary recrystallization grain so that the core loss thereof would be possibly unsatisfactory. The core loss value of such a grain-oriented electrical steel strip can be decreased remarkable by a magnetic domain control technology disclosed in, for example, U.S. Patent No. 4,770,720.

A grain-oriented electrical steel strip has a magnetic easy axis of <001> orientation in its rolling direction, and also has a {110} plane on its rolled surface.

The aggregation to {110}<001> orientation in electrical steel strip is achieved by utilizing a catastrophic phenomenon of grain growth called secondary recrystallization. The control of secondary recrystallization essentially requires the control of a primary recrystallization texture and structure prior to the secondary recrystallization thereof and the control of an inhibitor, i.e. a fine precipitate, or an element of the intergranular segregation type. The inhibitor inhibits the growth of any grains other than those having a {110}<001> orientation in the primary recrystallization texture and enables the selective growth of the grains having a {110}<001> orientation.

The following are the three typical processes which are known for the industrial manufacture of grain-oriented electrical steel strips or sheets:

(1) The process as disclosed by M.F. Littmann in U.S. Patent No. 2,599,340 (Japanese Patent Publication No. 3651/1955) which employs two steps of cold rolling utilizing MnS as the inhibitor;

(2) The process as disclosed by Taguchi and Sakakura in U.S. Patent No. 3,287,183 (Japanese Patent Publication No. 15644/1965) which adopts a reduction rate exceeding 80% in final cold rolling utilizing an inhibitor comprising A1N and MnS; and

(3) The process as disclosed by Imanaka et al. in U.S. Patent No. 3,932,234 (Japanese Patent Publication No. 13469/1976) which employs two steps of cold rolling utilizing an inhibitor comprising MnS (or MnSe) and Sb.

These processes have made it possible to produce on a commercial basis grain-oriented electrical steel strips having so high a degree of the aggregation to {110}<001> orientation that the strips have a magnetic flux density (B_8 value) of about 1.92 tesla.

In a process of manufacturing a grain-oriented electrical steel strip, various factors during the steps thereof affect upon the magnetic properties of products, and the conditions to the manufacture are seriously controlled during operation. Even with a such a seriously controlled manufacturing process, there are sometimes produced products having an inferior secondary recrystallization and bad magnetic properties about which the particular reason can't be found. In such a case, if occurrence of products having an inferior secondary recrystallization and bad magnetic properties can be known beforehand at upstream steps, a composition system and manufacturing conditions would be adjusted to make the secondary recrystallization satisfactory so as to produce a product in excellent in secondary recrystallization and in

magnetic properties.

However, although there have been made many trials to the forecast of occurrence of products having an inferior secondary recrystallization and bad magnetic properties up to now, no success have yet been achieved.

Summary of the Invention:

The present invention is devised in view of such a new finding by the inventors that the micro structure of a material after the step of annealing for decarburization but before the step of final annealing greatly affects upon the quality of secondary recrystallization and the magnetic properties of a product, and accordingly one object of the present invention is to provide a grain-oriented electrical steel strip having an extremely high magnetic flux density by controlling the primary recrystallization structure to a desired one.

According to the present invention, a cold-rolled electrical steel strip is controlled so as to change the temperature and the time during the annealing step for decarburization so as to have a micro structure having an average grain size $\bar{d}$ of greater than 15 µm, and a coefficient σ^* of deviation in grain size of less than 0.6 (standard deviation of a distribution normalized by the average grain size $\bar{d}$) between the annealing step for decarburization and the final annealing. Prior to the annealing step for decarburization, the material might be subjected to a final high-reduction cold-rolling step for applying a reduction ratio of greater than 80%, thereby it is possible to obtain a grain-oriented electrical steel strip more excellent in the magnetic properties.

A grain-oriented electrical steel strip according to the present invention is obtained by a manufacturing process comprising the steps of: casting molten metal obtained by a conventionally used steel manufacturing process, into an ingot with the use of a continuously casting process or an ingot making process and then forming a slab, as necessary, through a blooming process; hot-rolling said ingot or slab so as to obtain a hot-rolled steel strip; annealing said hot-rolled steel strip as necessary; cold-rolling said hot-rolled steel strip by one time or by more than two times between which an intermediate annealing step is included, so as to form a cold-rolled steel strip having a final thickness; and annealing said cold-rolled steel strip for decarburization and then final annealing.

The present inventors paid attention to the micro structure of a material after the above-mentioned annealing step for decarburization so as to study the relationship between the recrystallization structure of a strip after the annealing step for decarburization (which strip will be hereinafter denoted as "decarburization-annealed strip") and the magnetic properties (magnetic flux density) over a wide range with various kinds of view points, and found that the relationship therebetween is very close. The result of carried-out experiments from which the above-mentioned finding was obtained will be explained in detail hereinbelow:

Figs. 1 and 2 show affection upon the magnetic flux density (B_8 value) of a product by an average diameter ($\bar{d}$) of a primary recrystallization grain (d is the diameter of the circle with the same area as the grain has.) and a coefficient σ^* of deviation in the diameter of the recrystallization grain which were obtained by image-analyzing the micro structure of a decarburization-annealed strip that was observed by an optical microscope (over entire area in the strip-thicknesswise direction).

Further, Fig. 3 shows micro structures (strip-thicknesswise direction) of decarburization-annealed strips having average diameter ($\bar{d}$) of recrystallization grains and coefficients σ^* of deviation in the diameter of the recrystallization grains, which are different variously.

In the above-mentioned experiment, a slab consisting of 0.020 to 0.090 % (by weight) of C, 3.2 to 3.3 % of Si, 0.010 to 0.045 % of acid-soluble Al, 0.0030 to 0.0100 % of N, 0.0030 to 0.0300 % of S, 0.070 to 0.500 % of Mn and the balance of Fe and impurities, was heated up to 1,150 to 1,400 °C, and then was hot-rolled into a hot-rolled strip (hot strip) having a thickness of 2.3 mm. The hot-rolled strip was further subjected to a final high reduction cold-rolling step for applying a reduction ratio of about 88 % thereto after annealing the hot-rolled strip at a temperature in a temperature range of 900 to 1,200 °C so as to obtain a cold-rolled strip having a final thickness of 0.285 mm, and the thus obtained cold-rolled strip was annealed for decarburization at a temperature of a temperature range of 830 to 1,000 °C, and then was annealed after an annealing separation agent containing MgO as a main component being coated thereon.

As clearly understood from Figs. 1 and 2, it is possible to obtain a product having an extremely high magnetic flux density B_8 equal to or greater than 1.88 tesla with an average diameter $\bar{d}$ of primary recrystallization grain (decarburization-annealed strip) which is equal to or greater than 15 µm and with a coefficient σ^* of deviation in the grain diameter which is equal to or less than 0.6. Further, Figs. 1 and 2 show such a fact that a satisfactory recrystallization and a product having satisfactory magnetic properties can be obtained by setting the average diameter $\bar{d}$ of grain in a decarburization-annealed strip and the

coefficient σ^* of deviation in the diameter in suitable ranges.

The inventors have considered that causes affecting upon the relationship between the average diameter $\bar{d}$ of grain as well as the coefficient σ^* of deviation in the diameter of grain and occurrence of inferior secondary recrystallization or the magnetic flux density B_8 of a product are as follows although these are not always sure:

There are considered the micro structure (average diameter of grain, and distribution of grain diameters) of primary recrystallization, the texture, the inhibitor strength and the like as factors affecting upon the secondary recrystallization, including the orientation of secondary recrystallization.

Since variations in the texture and the distribution of diameters of grain occur along with the growth of grain after completion of primary recrystallization, the average diameter of the primary recrystallization grain indirectly exhibits the texture and the distribution of diameters of grain. The average diameter of grain in the decarburization-annealed strip itself is a value reversely proportional to the sum total of grain boundary areas (per unit area), and the intergranular energy give a drive force to the growth of grain in secondary recrystallization. Accordingly, the average diameter of grain in decarburization-annealed strip is considered as a parameter which is simultaneously descriptive of the texture, the distribution of grain diameters and the sum total of grain boundary areas which affect upon the secondary recrystallization.

The texture exhibits qualitative rates of crystal orientation ({110}<001> oriented grain or the like), oriented grain facilitating the grain growth of secondary recrystallization grain ({111}<112> oriented grain or the like) and other orientated grains, and further, the distribution of grain diameters affects upon the nucleation of secondary recrystallization grain, the ease of the grain growth and the nonuniformity of the grain growth. Accordingly, it can be considered that the average diameter $\bar{d}$ of grain in the decarburization-annealed strip which is a parameter simultaneously descriptive of the texture, the distribution of grain diameters and the sum total of grain boundary areas, has a strong correlation to the orientation of secondary recrystallization grain.

Meanwhile, the coefficient σ^* of deviation in the diameter of grain in the decarburization-annealed strip exhibits the nonuniformity of grain diameter, that is, the larger the coefficient σ^* of deviation in the diameter of grain, the harder the nucleation of secondary recrystallization grain and the growth of the grain become, and inferior secondary recrystallization seems to occur. Thus, the coefficient σ^* of deviation in the diameter of grain has a close relationship with occurrence of inferior secondary recrystallization, the average diameter $\bar{d}$ of grain in the decarburization-annealed strip has a close relationship with magnetic flux density of a product in the case of satisfactory secondary recrystallization. Accordingly, products having a high magnetic flux density (B_8) at a high yield rate can be manufactured by controlling the above-mentioned parameters in predetermined ranges:

Brief Description of the Drawings:

Fig. 1 is a graph indicating affection upon the magnetic flux density (B_8) of a product by an average diameter $\bar{d}$ of grain in a decarburization-annealed strip;

Fig. 2 is a graph indicating affection upon the magnetic flux density (B_8) of the product by the coefficient σ^* of deviation in the diameter of grain which is normalized by the average diameter $\bar{d}$ of grain in the decarburization-annealed strip;

Fig. 3 is a microscopic photograph view illustrating the micro structures of decarburization-annealed strips having variously different average diameters $\bar{d}$ of grain and coefficients σ^* of deviation in the diameter of grain;

Fig. 4 is a graph showing the relationship between the core loss value and the diameter of primary recrystallization grain in decarburization-annealed strip; and

Fig. 5 is a graph showing the relationship between the core loss value and the temperature of annealing for decarburization.

Best Preferred Embodiment Carrying out the Invention:

Explanation will be made hereinbelow of requisites of the present invention.

The components of a slab used in the present invention, although they should not be limited specifically, are very important in order to stabilize the magnetic properties of a product, and include preferably 0.025 to 0.100 % by weight of C and 2.5 to 4.5 % of Si. Further, Al, N, Mn, S, Se, Sb, B, Cu, Si, Nb, Cr, Sn, Ti and the like can be added as elements for forming an inhibitor.

The heated temperature of the slab is preferably less than 1,300°C in view of the energy cost although

it should not be limited specifically to this temperature. The heated slab is then hot-rolled into a hot-rolled strip. The hot-rolled strip after being annealed as necessary is then cold-rolled by one time or by more than two times between which an intermediate annealing is carried out, into a cold-rolled strip having a final thickness. The reduction ratio in the final cold-rolling step is preferably greater than 80 %, although it should not be limited specifically to this value, in order to increase the magnetic flux density (B_8) of the product. By setting the reduction ratio in the final cold-rolling step to greater than 80 %, it is possible to obtain, suitable amounts of {110}<001> orientated grain which is sharp and coincidence orientation grain ({111}<112> orientated grain or the like) which is likely eroded by the aforementioned orientated grain.

After the final cold-rolling step, the cold-rolled strip is annealed for decarburization, and is coated with an annealing separation agent containing MgO as a main component before it is formed into a strip coil which is then annealed.

According to the present invention, it is important to set the average diameter $\bar{d}$ of grain in the material before final annealing (primary recrystallization grain) to greater than 15 µm, and the coefficient σ^* of deviation in the diameter of grain is set to less than 0.6.

In order to obtain a decarburization-annealed strip having such a micro structure, it is possible to employ, for example, a process of adjusting the number of primary recrystallization nucleuses by a reduction ratio in cold-rolling, grain diameters in a material to be cold-rolled and the like, a process of controlling the grain growth during annealing for decarburization in which the strength of the inhibitor during annealing for decarburization is adjusted by manipulating the content amounts of elements forming the inhibitor, the heating temperature of the slab, the coiling temperature of the strip after hot-rolling, the temperature of annealing of the hot-rolled strip or the like, a process of controlling the grain growth by adjusting the temperature and time of the annealing for decarburization, and the like.

Further, it is possible to set the average diameter $\bar{d}$ of material grain before final annealing (primary recrystallization grain) to greater than 15 µm, and the coefficient σ^* of deviation in the diameter of grain to less than 0.6 by additionally annealing the material between the steps of annealing for decarburization and final annealing.

In order to on-line measure the diameter of primary recrystallization grain in the material (strip) during or after annealing for decarburization, a core loss value is measured by passing the strip between primary and secondary core loss measuring coils, and then the diameter of primary recrystallization grain in the material is detected with the use of the relationship shown in Fig. 4. With the use of such a grain diameter measuring means, the diameter of primary recrystallization grain is on-line measured, and then feed-back or feed-forward control for changing the temperature and time of annealing for decarburization are carried out so as to set the diameter of primary recrystallization grain to greater than 15 µm. Fig. 5 shows the relationship between the temperature obtained during annealing for decarburization and the diameter of primary recrystallization grain. Although the diameter of primary recrystallization grain is indicated with the use of a core loss value of a decarburization-annealed strip, the diameter of primary recrystallization grain can be known from Fig. 5 with the use of the relationship between the core loss value and the diameter of primary recrystallization grain in the decarburization-annealed strip, shown in Fig. 4.

The result shown in Fig. 5 gives the diameters of primary recrystallization grain (Fig. 5 shows corresponding core loss values) which are obtained by variously changing the temperature of annealing while the time of annealing is fixed to 150 sec. For example, the diameter of primary recrystallization grain can be also controlled by changing the duration of annealing (strip feeding ratio) while fixing the annealing temperature to, for example, 850° C.

As to the composition and coating amount of the annealing separation agent, the final annealing and the like, there are applied no specific conditions in particular. However, in order to prevent a suitable micro structure of the decarburization-annealed strip from being turned into an unsuitable micro structure with grain growth during an increase in temperature of final annealing, the material after primary recrystallization is preferably to nitrided in the atmosphere in which NH_3 having a concentration of higher than 1,000 ppm is mixed in mixture gas containing hydrogen gas and nitrogen gas while oxidation potential P_{H2O}/P_{H2} being set to be equal to or less than 0.04 under a temperature range from 500 to 900° C and under, for example, such a condition that the strip is made to run, thereby to heighten the strength of the inhibitor during increase in temperature of final annealing, in view of stable manufacture of electrical steel strips. Sulphurizing treatment can be also used for increasing the strength of the inhibitor. Further, although the strength of the inhibitor should be lowered during annealing for decarburization in order to obtain a desired primary recrystallization structure by annealing for decarburization in a relative low temperature range (less than 800° C), if the strength of the inhibitor is insufficient to stably carry out secondary recrystallization, it is required during final annealing to heighten the above-mentioned inhibitor strength. As to an example of inhibitor strengthening processes, there has been known a process of setting the partial pressure of

nitrogen in atmospheric gas for final annealing to a comparatively high value with steel containing Al.

The reason why the average diameter $\bar{d} \geq$ 15 µm and the coefficient σ^* ≤ 0.6 of deviation in the diameter of grain are provided, is such that a product having a satisfactory magnetic flux density B_8 of greater than 1.88 tesla can be obtained when the average diameter $\bar{d}$ and the coefficient σ^* of deviation in the diameter of grain fall in the above-mentioned ranges, as is clear from Figs. 1 and 2. It is noted that although the upper limit of the average diameter $\bar{d}$ is not defined in particular, the upper limit of the average diameter $\bar{d}$ is 50 µm in view of the condition of usual components and the conditions of steps. More than 50 µm of average diameter $\bar{d}$ is unpreferable since a cost-up is caused due to higher purities of the components, a rise in annealing temperature and the like. On the other hand, the coefficient σ^* of deviation in the diameter of grain is allowed to have a value down to zero.

As mentioned above, the reason why the condition of primary recrystallization grain before final annealing is provided, is such that satisfactory magnetic properties can be obtained by additional heat-treatment after annealing for decarburization and before final annealing so as to adjust the average diameter $\bar{d}$ of primary recrystallization grain to a value equal to or greater than 15 µm and the coefficient σ^* of deviation in the diameter of grain to a value equal to or less than 0.6.

--Reference Example 1--

A slab containing 0.054 wt.% of C, 3.25 wt.% of Si, 0.15 wt.% of Mn, 0.005 wt.% of S, 0.027 wt.% of acid-soluble Al and 0.0078 wt.% of N was heated up to 1,150°C, and was then hot-rolled into a hot-rolled strip having a thickness of 2.3 mm. This hot-rolled strip was annealed at a temperature of 1,150°C and 900°C, and thereafter was cold-rolled at a cold-rolling reduction of about 88 % into a cold-rolled strip having a final thickness of 0.285 mm. The cold-rolled strip was held at a temperature of 810°C for 150 sec. and was then annealed for decarburization at a temperature of 830°C, 890°C and 950°C for 20 sec., respectively. The thus obtained decarburization-annealed strip was coated thereover with annealing separation agent having MgO as a main component, and was heated up to 1,200°C at a rate of 10°C/hour in the atmospheric gas containing 25 % of N_2 and 75 % of H_2, and was then held at a temperature of 1,200°C for 20 hours in the atmospheric gas containing 100 % of H_2 in order to carry out final annealing.

After annealing for decarburization, the average diameter $\bar{d}$ and the coefficient σ^* of deviation in the diameter of grain of the decarburization-annealed strip were measured with the use of an image analyzer. Table 1 shows the results of the image analysis and the magnetic properties of products.

TABLE 1

Anneal Temp. of Hot-Rolled Strip (°C)	Anneal Temp. for Decarbu. (°C)	Average Dia. $\bar{d}$ (µm)	Deviat. Coeffi. in Dia. σ^*	Magnetic Flux Density B_8 (T)	Second. Recryst. Rate (%)	Remark
1,150	830	13	0.45	1.85	100	C.E.
1,150	890	19	0.48	1.92	100	P.I.
1,150	950	23	0.53	1.92	100	P.I.
900	830	18	0.47	1.92	100	P.I.
900	890	23	0.52	1.93	100	P.I.
900	950	30	0.62	1.68	30	C.E.
Note: C.E. = Comparison Example,						
P.I. = Present Invention						

--Reference Example 2--

A slab containing 0.058 wt.% of C, 3.28 wt.% of Si, 0.14 wt.% of Mn, 0.007 wt.% of S, 0.025 wt.% of

acid-soluble Al and 0.0075 wt.% of N was heated up to a temperature of 1,150 °C or 1,250 °C, and then was hot-rolled into a hot-rolled strip having a thickness of 2.3 mm. This hot-rolled strip was held at a temperature of 1,150 °C for 30 sec., and was then held at a temperature of 900 °C for 30 sec. in order to be annealed. Then, the hot-rolled strip was cold-rolled at a reduction ratio of about 88% into a cold-rolled strip having a final thickness of 0.285 mm, which was then annealed for decarburization by being held at a temperature of 850 °C for 150 sec.

The thus obtained decarburization-annealed strip is coated thereover with annealing separation agent containing MgO as a main component, heated up to 1,200 °C at a rate of 10 °C/hour in the atmospheric gas containing 25 % of N_2 and 75 % of H_2, and was then held at a temperature of 1,200 °C for 20 hours in the atmospheric gas containing 100 % of H_2 for final annealing.

After annealing for decarburization, the average diameter $\bar{d}$ and the coefficient σ^* of deviation in the diameter of grain of the decarburization-annealed strip were measured with the use of an image analyzer. Table 2 shows the results of the image analysis and the magnetic properties of products.

TABLE 2

Slab Heating Temp. (°C)	Average Dia. $\bar{d}$ (µm)	Deviat. Coeffi. in Dia. σ^*	Magnetic Flux Density B_8 (T)	Secondary Recryst. Rate (%)	Remarks
1,150	21	0.49	1.93	100	P.I.
1,250	14	0.44	1.87	100	C.E.

--Reference Example 3--

The decarburization-annealed strip obtained at a slab heating temperature of 1,250 °C in the conditions mentioned in the reference example 2, was heat-treated at a temperature of 950 °C for 30 sec., coated with annealing separation agent containing MgO as a main component, and then was annealed under the conditions mentioned in the reference example 2.

Table 3 shows the average diameter $\bar{d}$ and diameter deviation coefficient σ^* of the steel strip (entire thickness in cross-section) together with the magnetic flux density B_8 of the product.

TABLE 3

Presence of Additional Heat-Treat.	Average Dia. $\bar{d}$ (µm)	Deviat. Coeffi. in Dia. σ^*	Magnetic Flux Density B_8 (T)	Secondary Recryst. Rate (%)	Remarks
No	14	0.45	1.87	100	C.E.
Yes	18	0.49	1.92	100	P.I.

--Reference Example 4--

A slab containing 0.056 wt.% of C, 3.27 wt.% of Si, 0.14 wt.% of Mn, 0.008 wt.% of S, 0.027 wt.% of acid-soluble Al and 0.0078 wt.% of N was heated up to a temperature of 1,150 °C and then was hot-rolled into a hot-rolled strip having a thickness of 2.0 mm. The hot-rolled strip was held at 1,120 °C for 30 sec., then held at 900 °C for 30 sec., for annealing, and was then cold-rolled at a reduction ratio of about 89 % into a cold-rolled strip having a final thickness of 0.220 mm, which was held at 830 °C for 90 sec. and was then annealed for decarburization at a temperature of 890 °C and 920 °C for 20 sec., respectively. The thus obtained decarburization-annealed strip was coated thereover with annealing separation agent containing

MgO as a main component, heated up to 880 °C in the atmospheric gas containing 25 % of N_2 and 75 % of H_2, and then heated up to 1,200 °C from 880 °C in the atmospheric gas containing 75 % of N_2 and 25 % of H_2, and was then held at 1,200 °C for 20 hours in the atmospheric gas containing 100 % of H_2 for final annealing. At this time, the rate of temperature rise up to 1,200 °C was set to 10 °C/hour and 25 °C/hour.

After annealing for decarburization, the average diameter $\bar{d}$ and diameter deviation coefficient σ^* of the decarburization-annealed strip (entire thickness in cross-section) were measured with the use of an image analyzer. Table 4 shows the conditions of heat-treatment, the results of the image analysis and the magnetic properties.

TABLE 4

Anneal Temp. for Decarbu. (°C)	Temp. Rise. Rate (°C/hr)	Average Dia. $\bar{d}$ (µm)	Deviat. Coeffi. in Dia. σ^*	Magnetic Flux Density B_8 (T)	Second. Recryst. Rate (%)	Remark
890	10	22	0.55	1.94	100	P.I.
890	25	22	0.55	1.93	100	P.I.
920	10	25	0.61	1.73	52	C.E.
920	25	25	0.61	1.70	40	C.E.

--Reference Example 5--

The decarburization-annealed strip obtained under the conditions mentioned in the reference example 4, was coated thereover with annealing separation agent containing MgO as a main component, heated up to 1,200 °C at a heating rate of 15 °C/hour in the atmospheric gas of containing 25 % of N_2 and 75 % of H_2 and in the atmospheric gas containing 50 % of N_2 and 50 % of H_2, and was then held at 1,200 °C for 20 hours in the atmospheric gas containing 100 % of H_2 for final annealing.

After annealing for decarburization, the average diameter $\bar{d}$ and the diameter deviation coefficient σ^* of the decarburization-annealed strip were measured with the use of an image analyzer. Table 5 shows the conditions of treatment, the results of the image analysis and the magnetic properties of products.

TABLE 5

Anneal Temp. for Decarbu. (°C)	Atmospheric Gas N_2/H_2	Average Dia. $\bar{d}$ (µm)	Deviat. Coeffi. in Dia. σ^*	Magnetic Flux Density B_8 (T)	Second Recryst. Rate (%)	Remark
890	25/75	22	0.55	1.93	100	P.I.
890	50/50	22	0.55	1.92	100	P.I.
920	25/75	25	0.61	1.71	43	C.E.
920	50/50	25	0.61	1.79	58	C.E.

--Reference Example 6--

A slab containing 0.045 wt.% of C, 3.20 wt.% of Si, 0.065 wt.% of Mn, 0.023 wt.% of S, 0.08 wt.% of Cu and 0.018 wt.% of Sb was heated up to 1,300 °C, and was thereafter hot-rolled into a hot-rolled strip having a thickness of 2.6 mm. This hot-rolled strip was held at 900 °C for three minutes for annealing, and was then cold-rolled at a reduction ratio of about 63 % into a cold-rolled strip having a thickness of 0.95 mm,

and was held at 950 °C for three minutes for intermediate annealing. Then the cold-rolled strip was cold-rolled at a reduction ratio of 70 % so as to have a final thickness of 0.285 mm, and was held 810 °C, 850 °C and 890 °C for 200 sec., respectively, in order to be annealed for decarburization. The thus obtained decarburization-annealed strip was coated thereover with annealing separation agent containing MgO as a main component, was heated up to 1,200 °C at a rate of 5 °C/hour in the atmospheric gas containing 25 % of N_2 and 75 % of H_2, and then was held at 1,200 °C for 20 hours in the atmospheric gas containing 100 % of H_2 for final annealing.

After annealing for decarburization, the average diameter $\bar{d}$ and diameter deviation coefficient σ^* of the decarburization-annealed strip (entire thickness in cross-section) were measured with the use of an image analyzer. Table 6 shows the conditions of treatment, the results of the image analysis and the magnetic properties of products.

TABLE 6

Anneal Temp. for Decarbu. (°C)	Average Dia. $\bar{d}$ (μm)	Deviat. Coeffi. in Dia. σ^*	Magnetic Flux Density B_8 (T)	Secondary Recryst. Rate (%)	Remarks
810	14	0.55	1.84	100	C.E.
850	16	0.57	1.88	100	P.I.
890	18	0.63	1.75	71	C.E.

--Reference Example 7--

A slab containing 0.05 % by weight of C, 3.25 % of Si, 0.028 % of acid-soluble Al, 0.0075 % of N, 0.007 % of S and 0.014 % of Mn was heated up to 1,150 °C, and was hot-rolled in a conventional manner so as to obtain a hot-rolled strip having a thickness of 1.8 mm.

Then the hot-rolled strip was annealed at 1,150 °C, and was cold-rolled into a cold-rolled strip having a thickness of 0.19 mm after pickling, which was then slitted into test pieces having a width of 60 mm, and core loss thereof were on-line measured in an experimental continuous annealing furnace. The annealing was carried out by changing the annealing temperature in a range from 810 to 870 °C and the annealing duration in a range from 90 to 150 sec. in the atmosphere of 75 % of H_2 and 25 % of N_2 having a dew point of 55 °C.

Fig. 4 shows the relationship between the core loss value $W_{14/50}$ of the steel strip and the average grain diameter of a test pieces taken out from a part of the core loss measuring section. That is, it can be understood that the average diameter $\bar{d}$ (μm) can be obtained from the core loss value W (W/kg) with the use of the following formula (1) within a degree of accuracy of +/- 1 μm:

$\bar{d} = -11.17 W + 52.33$ (μm) (1)

Further, it can be understood in comparison with Fig. 1 that the above-mentioned degree of accuracy is sufficient to ensure a high magnetic flux density while preventing inferior secondary recrystallization.

Advantages of the Invention:

As mentioned above, according to the present invention, the average grain diameter $\bar{d}$ and diameter deviation coefficient σ^* of primary recrystallization grain before final annealing are controlled so as to stably manufacture a grain-oriented electrical steel strip having excellent magnetic properties. Further, the average diameter $\bar{d}$ and the diameter deviation coefficient σ^* can be used as parameters for forecasting the magnetic flux density of the product, and therefore, the magnetic flux density of the product can be set to a desired value by adjusting, for example, the conditions of final annealing.

Claims

1. A process of manufacturing a grain-oriented electrical steel strip, in which a slab containing 0.025 to 0.100 % by weight of C, 2.5 to 4.5 % of Si, if necessary one or more than two elements which form inhibitor and the balance of Fe in essential is hot-rolled and cold-rolled by one time or more than two times between which an intermediate annealing step is carried out, into a cold-rolled strip having a final thickness, and then said cold-rolled strip is annealed for primary recrystallization (annealing for decarburization) after said cold-rolled strip being coated with annealing separation agent, characterized in that the average diameter of said material before final annealing is set to a value greater than 15 µm and the coefficient σ^* of deviation in grain diameter is set to a value less than 0.6 by use of at least one of the steps of: adjusting the number of primary recrystallization nucleuses by controlling the reduction ratio in cold-rolling or the diameter of grain in the material to be cold-rolled; adjusting the strength of inhibitor by controlling at least one of the content of an element which forms said inhibitor, the heating temperature of said slab, the coiling temperature of the strip after hot-rolling, and the annealing temperature of the hot-rolled strip; adjusting the temperature and time of annealing for decarburization; and additionally annealing the material between annealing for decarburization and final annealing.

2. A process of manufacturing a grain-oriented electrical steel strip, in which a slab containing 0.025 to 0.100 % by weight of C, 2.5 to 4.5 % of Si, if necessary one or more than two elements which form inhibitor and the balance of Fe in essential is hot-rolled and cold-rolled by one time or more than two times between which an intermediate annealing step is carried out, into a cold-rolled strip having a final thickness, and then said cold-rolled strip is annealed for primary recrystallization (annealing for decarburization) after said cold-rolled strip being coated with annealing separation agent, characterized in that the diameter of grain of the material (strip) is on-line measured after completion of primary recrystallization during said annealing step for decarburization, and feed-back or feed-forward control for changing the temperature and time of annealing during said annealing step for decarburization is carried out in accordance with the result of the measurement.

3. A process of manufacturing a grain-oriented electrical steel strip, in which a slab containing 0.025 to 0.100 % by weight of C, 2.5 to 4.5 % of Si, if necessary one or more than two elements which form inhibitor and the balance of Fe in essential is hot-rolled and cold-rolled by one time or more than two times between which an intermediate annealing step is carried out, into a cold-rolled strip having a final thickness, and then said cold-rolled strip is annealed for primary recrystallization (annealing for decarburization) after said cold-rolled strip being coated with annealing separation agent, characterized in that the average diameter of said material before final annealing is set to a value greater than 15 µm and the coefficient σ^* of deviation in grain diameter is set to a value less than 0.6 by use of at least one of the steps of: adjusting the number of primary recrystallization nucleuses by controlling the reduction ratio in cold-rolling or the diameter of grain in the material to be cold-rolled; adjusting the strength of inhibitor by controlling at least one of the content of an element which forms said inhibitor, the heating temperature of said slab, the coiling temperature of the strip after hot-rolling, and the annealing temperature of the hot-rolled strip; adjusting the temperature and time of annealing for decarburization; and additionally annealing the material between annealing for decarburization and final annealing, and the material after primary recrystallization is nitrided in accordance with a diameter of grain (primary recrystallization grain) before final annealing.

FIG. 1

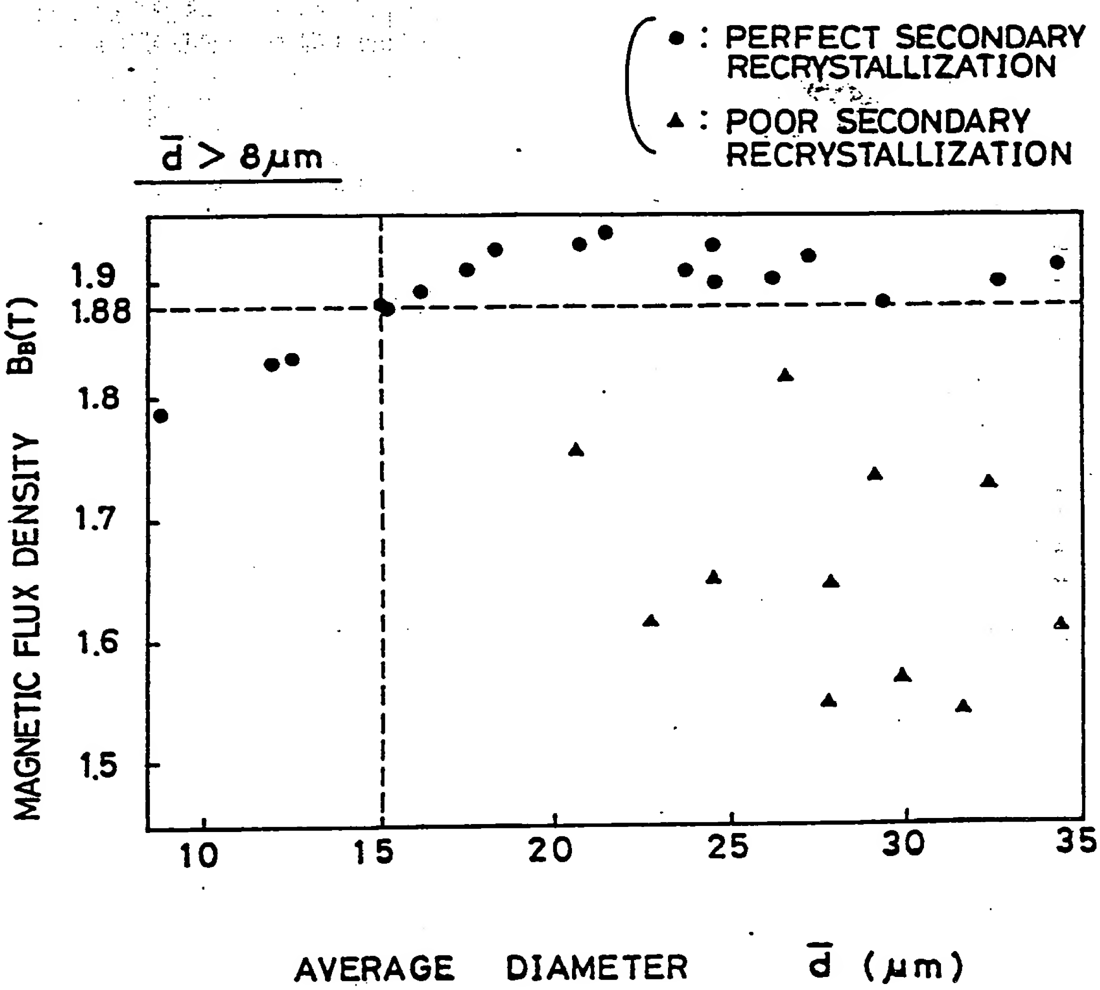

●: PERFECT SECONDARY RECRYSTALLIZATION
▲: POOR SECONDARY RECRYSTALLIZATION
$\bar{d} > 8\mu m$
MAGNETIC FLUX DENSITY B_B(T)
1.9
1.88
1.8
1.7
1.6
1.5
10
15
20
25
30
35
AVERAGE DIAMETER $\bar{d}$ (μm)

FIG. 2

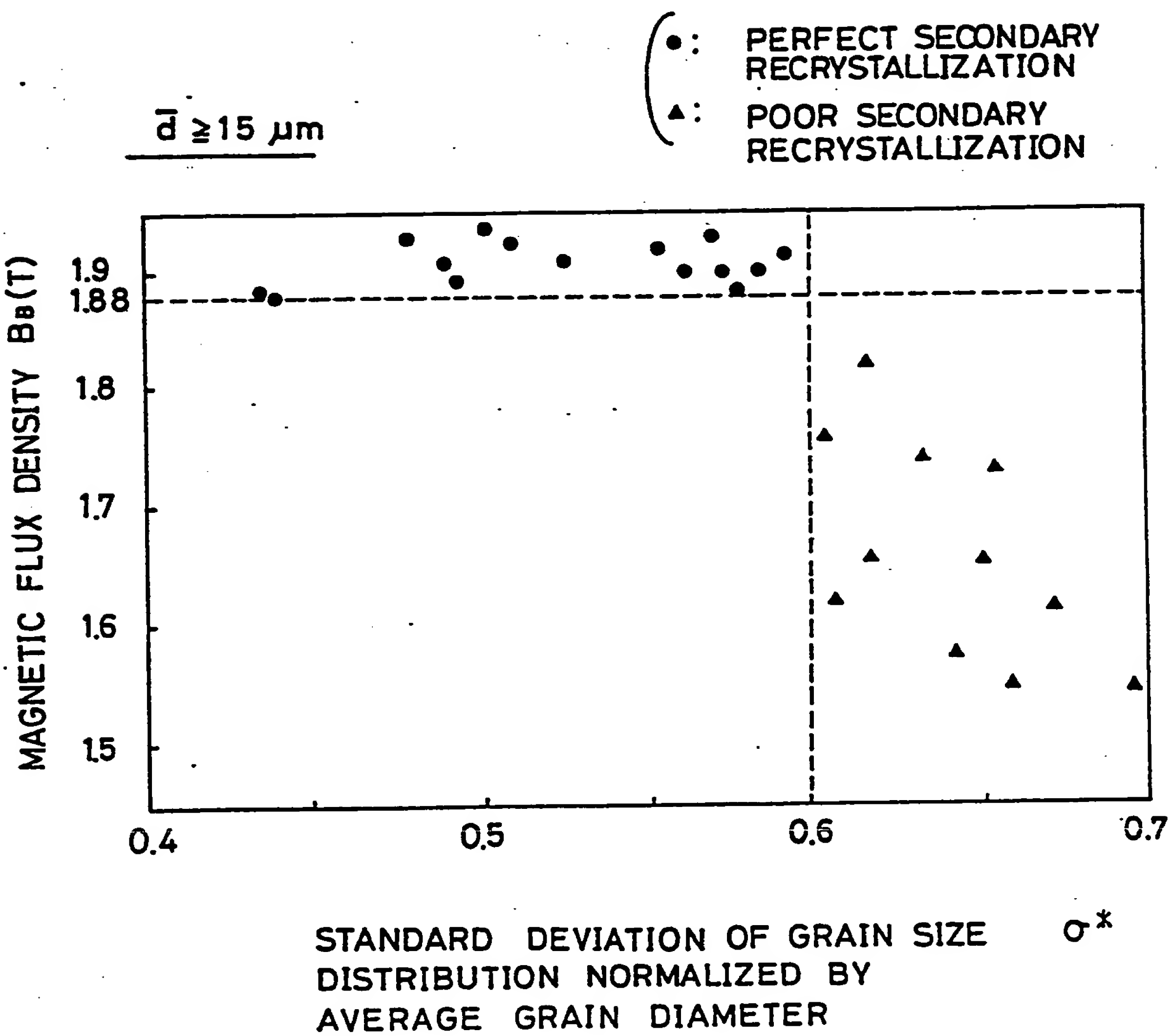

•: PERFECT SECONDARY RECRYSTALLIZATION
▲: POOR SECONDARY RECRYSTALLIZATION
d̄ ≥15 µm
MAGNETIC FLUX DENSITY B8(T)
1.9
1.88
1.8
1.7
1.6
1.5
0.4
0.5
0.6
0.7
STANDARD DEVIATION OF GRAIN SIZE DISTRIBUTION NORMALIZED BY AVERAGE GRAIN DIAMETER σ*

FIG. 3

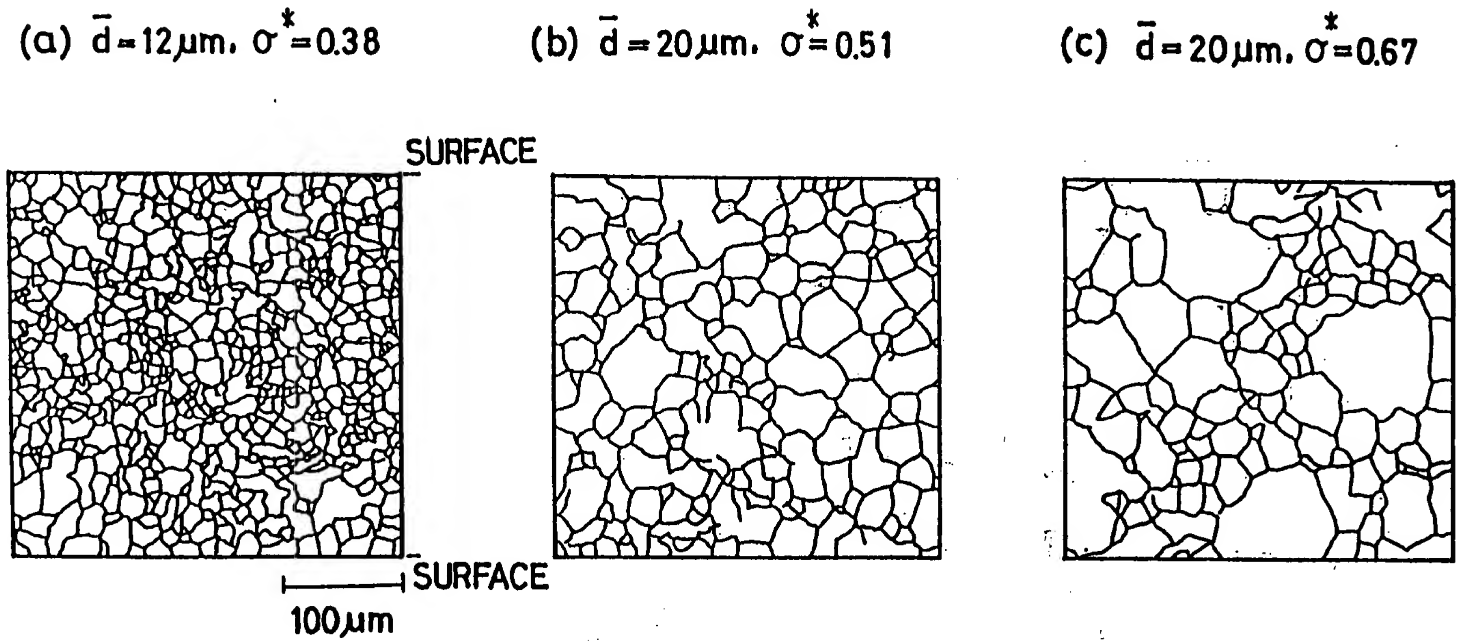

(a) $\bar{d}$=12μm, σ*=0.38
(b) $\bar{d}$=20μm, σ*=0.51
(c) $\bar{d}$=20μm, σ*=0.67
SURFACE
SURFACE
100μm

FIG. 4

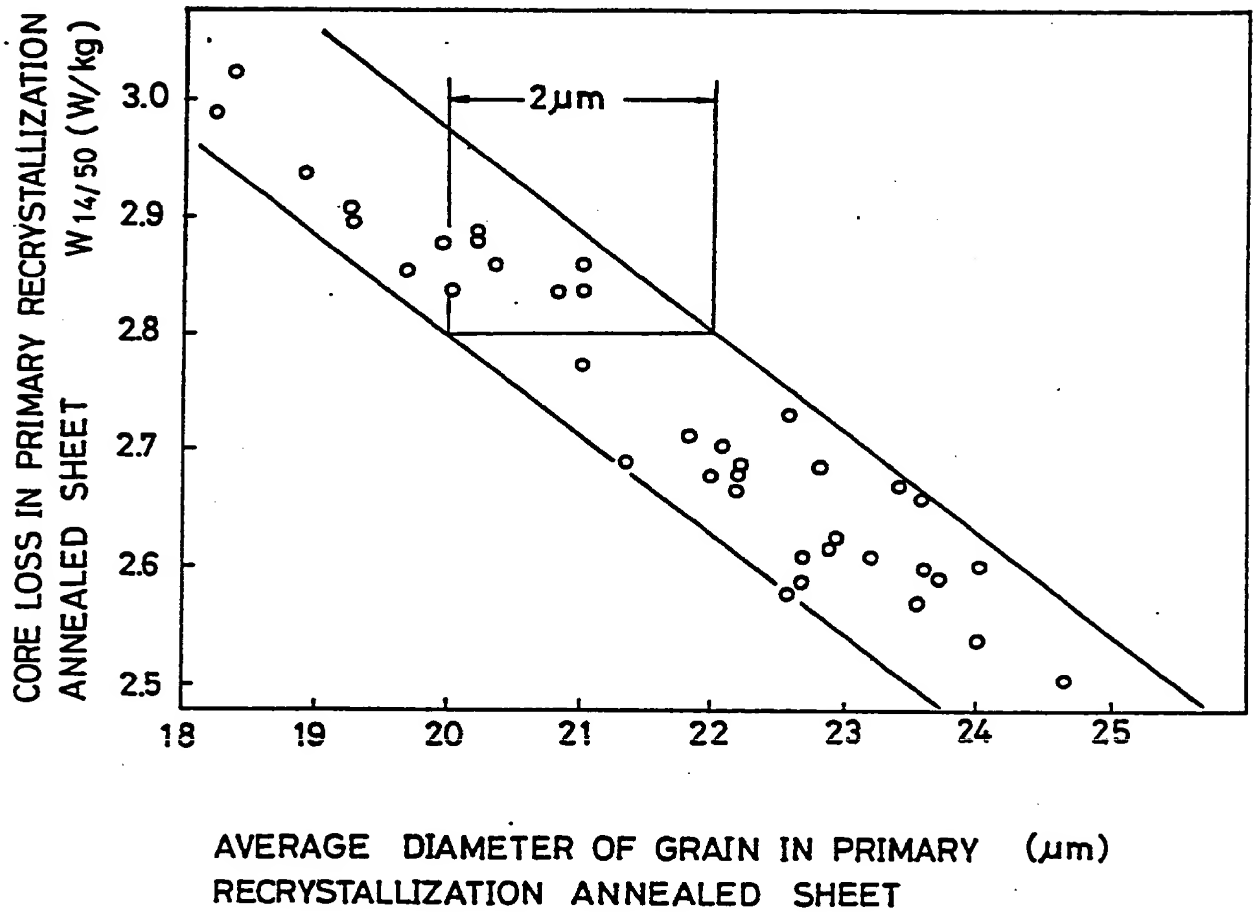
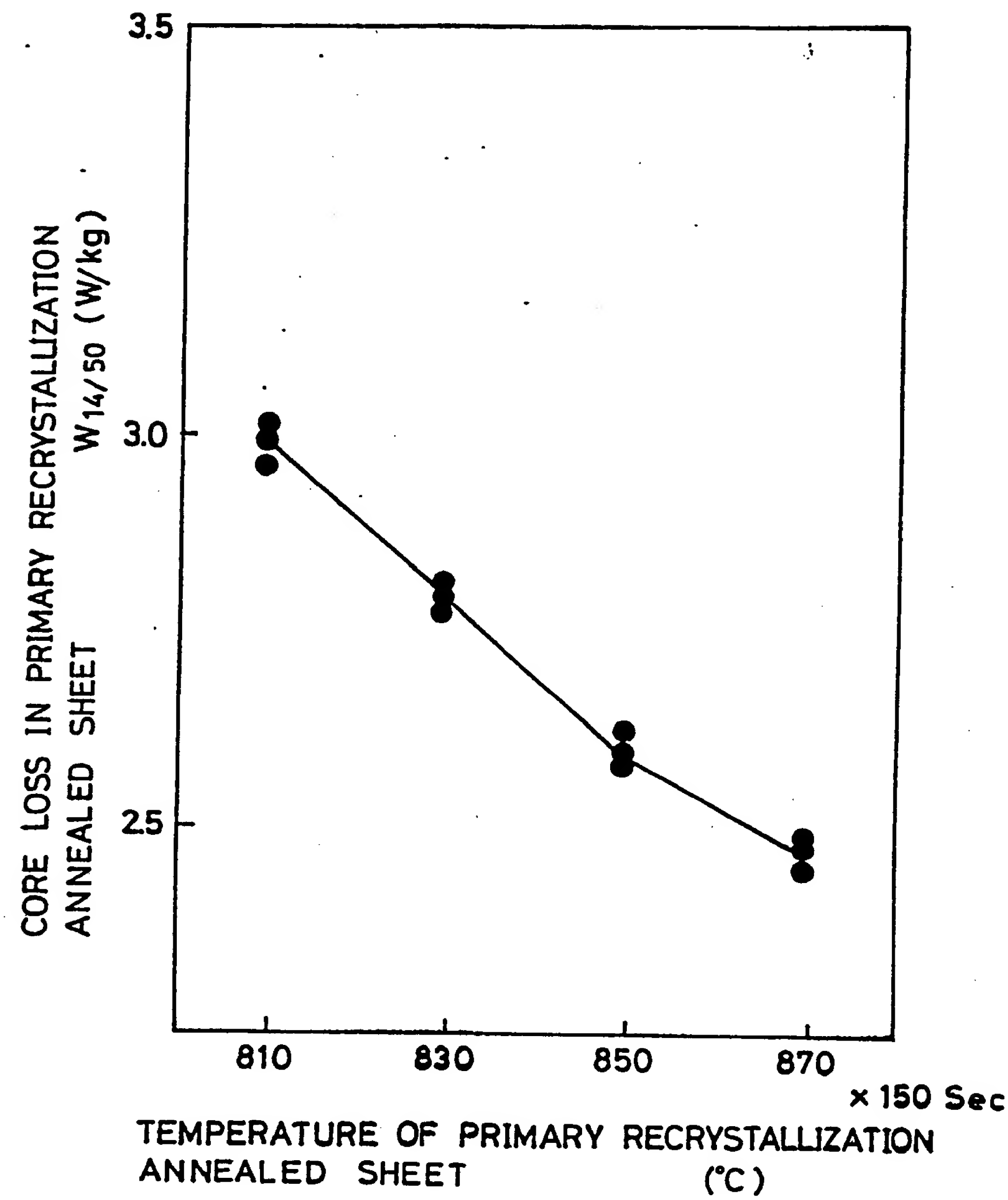

CORE LOSS IN PRIMARY RECRYSTALLIZATION ANNEALED SHEET W14/50 (W/kg)
3.0
2.9
2.8
2.7
2.6
2.5
2μm
18
19
20
21
22
23
24
25
AVERAGE DIAMETER OF GRAIN IN PRIMARY RECRYSTALLIZATION ANNEALED SHEET (μm)

FIG. 5


3.5
3.0
2.5
CORE LOSS IN PRIMARY RECRYSTALLIZATION ANNEALED SHEET $W_{14/50}$ (W/kg)
810
830
850
870
× 150 Sec
TEMPERATURE OF PRIMARY RECRYSTALLIZATION ANNEALED SHEET (°C)